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A mixture of massive and feathery microstructures of Ti48Al2Cr2Nb alloy by high undercooled solidification☆

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ABSTRACT

A mixture of massive and feathery microstructures was observed in Ti48Al2Cr2Nb alloy subjected to the undercooled solidification rather than the heat treatments in most cases. Double recalescence events and primary β solidification confirmed that massive γ phase did not directly nucleate from the undercooled melt but formed during the solid-state transformations. It is believed that small white areas (aluminium-poor) along lamellar grain boundaries may be closely related to the formation of massive γ phase and feathery γ phase. High dislocation density and stacking faults were detected in massive γ phase by transmission electron microscopy. The high energy of defects and undercooling in the solid state phase transformation can provide sufficiently high driving force for the nucleation of massive γ phase.

1. Introduction

Massive transformation in γ-TiAl alloys was first observed by Wang et al. in 1992 through cooling from the α-phase field at different cooling rates [1] and has attracted much attention since then [2–4]. It is now generally admitted that proper composition, proper cooling rate and proper grain size are required to give rise to massive transformation [5]. Subsequently, lamellar or massive γ phase (γ_m) was found by Z. G. Liu et al. at matrix α_2 grain boundaries in rapidly solidified Ti46Al2Cr2Nb alloy (a grain size of about 5–10 μm) with low cooling rate (9.9×10^5 K/s) and rapidly solidified Ti46Al2Cr2Nb1.0Y alloy (a grain size of about 0.5–3 μm) at high cooling rate (1.8×10^6 K/s), respectively [6]. Under non-equilibrium conditions, conventionally, such high cooling rate (10^5 K/s–10^6 K/s) is uncommon, but γ_m and feathery γ phase (γ_f) may occur at the edge of as-cast ingots or the sheet of TiAl based alloys because of the chilling by mould. The metastable microstructure has a noticeable influence on the quality of ingots or castings. γ_m was also discovered by O. Shuleshova et al. through substrate quenching of highly undercooled Ti50Al10Nb alloy and it was considered that γ_m was formed due to sluggish precipitation of the γ plates in the α matrix with increasing the Nb content [7]. It is well known that the effect of heavy alloying extended the massive transformation regime to lower cooling rate side in TiAl alloys [8,9]. However, high undercooled solidification is also an effective way to realize rapid solidification of TiAl based alloys at a relativity low cooling rate, obtaining stable microstructure or metastable microstructure [10]. A bulk of investigations focused on the heat treatments of γ_m and γ_f. Little attention was paid to a mixture of γ_m and γ_f by undercooled solidification and the detailed massive transformation in undercooled TiAl alloys is still uncertain yet at present.

In this work, a mixture of γ_m and γ_f was observed in the high undercooled solidification. Furthermore, the objectives were to confirm the primary β solidification at high undercooling in Ti48Al2Cr2Nb alloy and to prove that γ_m is not the primary phase ($L \rightarrow \gamma_{metastable}$). In addition, defects such as dislocations and stacking faults besides undercooling in the solid state phase transformation can provide enough driving force for the nucleation of γ_m at a low cooling rate.

2. Experimental

The alloy with the nominal chemical composition Ti48Al2Cr2Nb was prepared from Ti, Al, Cr and Ti52.47Nb (wt %) with 99.99% purity (or better) in an electromagnetic cold-crucible melting facility under Ar atmosphere. As-cast ingots were homogenized in a vacuum heat treatment furnace at 1080 K for 5 h. Samples of about 1–2 g mass were cut from the master ingots, polished and processed in an electromagnetic levitation apparatus. The vacuum chamber was evacuated to 10^{-4} Pa and backfilled to 0.05 MPa with purified Ar gas. The levitated sample was melted repeatedly, overheated by 100–150 K and cooled by purified He gas streaming across the surface. Thermal history data were obtained using a two-colour infrared pyrometer (ZX 100B) with an accuracy of ±5 K.

The sample was sectioned along direction perpendicular to the substrate and etched using 10 mL HNO_3 + 10 mL HF + 80 mL H_2O. The samples by undercooled solidification were characterized by scanning electron microscope (TESCAN VEGA3) (SEM), optical microscope

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Table 1
Chemical composition of the small white areas (Al-poor), B2, γ_m and α_2 observed in the undercooled microstructure of Ti48Al2Cr2Nb alloy (atomic percent).

Phase/areas	Ti	Al	Cr	Nb
Small white areas (Al-poor)	48.40	46.32	2.89	2.39
B2 (inside lamellar grains)	48.48	45.42	3.68	2.42
γ_m	47.46	48.39	1.77	2.63
α_2	48.00	47.85	1.84	2.31

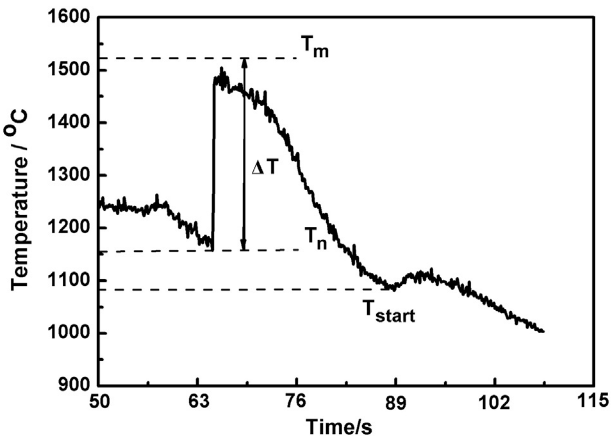

Fig. 1. Thermal history of the recalescence behaviour recorded by a two-colour pyrometer in undercooling experiments for Ti48Al2Cr2Nb alloy at $\Delta T = 370$ K.

(OLYMPUS GX71, Japan) (OM) and energy dispersive spectroscope (EDS). Each value given in Table 1 is the average value of at least 10 measurements in a same area. Thin foils for transmission electron microscopy (TEM) were thinned manually to the thickness of 50 μm and then made electron-transparent by ion-milling for microstructure observation using a TEM (FEI Tecnai G2 F30, America) operating at 200 kV. Constituent phases of the alloy were determined by X-ray diffraction (XRD) analyses using Cu Kα radiation.

3. Results and discussion

The recalescence event at $\Delta T = 370$ K for Ti48Al2Cr2Nb alloy is presented, revealed the different solidification behaviours and the relevant process stages are indicated in Fig. 1. The melting temperature is 1525 °C for Ti48Al2Cr2Nb alloy. The bulk undercooling (ΔT) is defined as the difference between the liquidus temperature (T_m) and the nucleation temperature (T_n) of the event [11]. The nucleation of primary β phase (~1155 °C) is represented by the steep recalescence (the melt undercooling below the liquidus temperature) in Fig. 1. After the first recalescence peak about a few microseconds, the duration time of post-recalescence period ($\Delta t_{pl} = \sim 5$ s), straight-forward cooling curve was obtained. It was concluded that the residual liquid transformed into solid after recalescence [12]. Subsequently, at cooling rate of ~11.5 K/s, a sudden $\alpha \rightarrow (\gamma_{Lamellar} + \gamma_m)$ transformation caused the second heat release at the temperature of ~1080 °C (T_{start}).

Fig. 2(a), (b) and (c) (d) shows the mixture of γ_m, γ_f and lamellar microstructures in Ti48Al2Cr2Nb alloy at high undercooling levels. Fig. 2(a) depicts the formation of diffusion-related feathery microstructure along lamellar grain boundaries at $\Delta T = 350$ K. A fine lamellar structure and γ_f co-existing with few γ_m along lamellar grain boundaries are observed by BSE in the high magnification, as shown in Fig. 2(c). With increasing undercooling, Fig. 2(b) shows more γ_m grains together with some γ_f mostly at lamellar grain boundaries and the details in the higher magnification by BSE are given in Fig. 2(d). The X-ray diffraction patterns for $\Delta T = 350$ K and $\Delta T = 370$ K display α_2 and γ phase reflections without B2 phase reflections in Fig. 3. The intensity of the γ phase peaks increases at $\Delta T = 370$ K, indicating the massive γ phase occurs. It gives evidence that γ_m grains are formed at $\Delta T = 370$ K. However, it is likely to form metastable γ phase from deeply undercooled

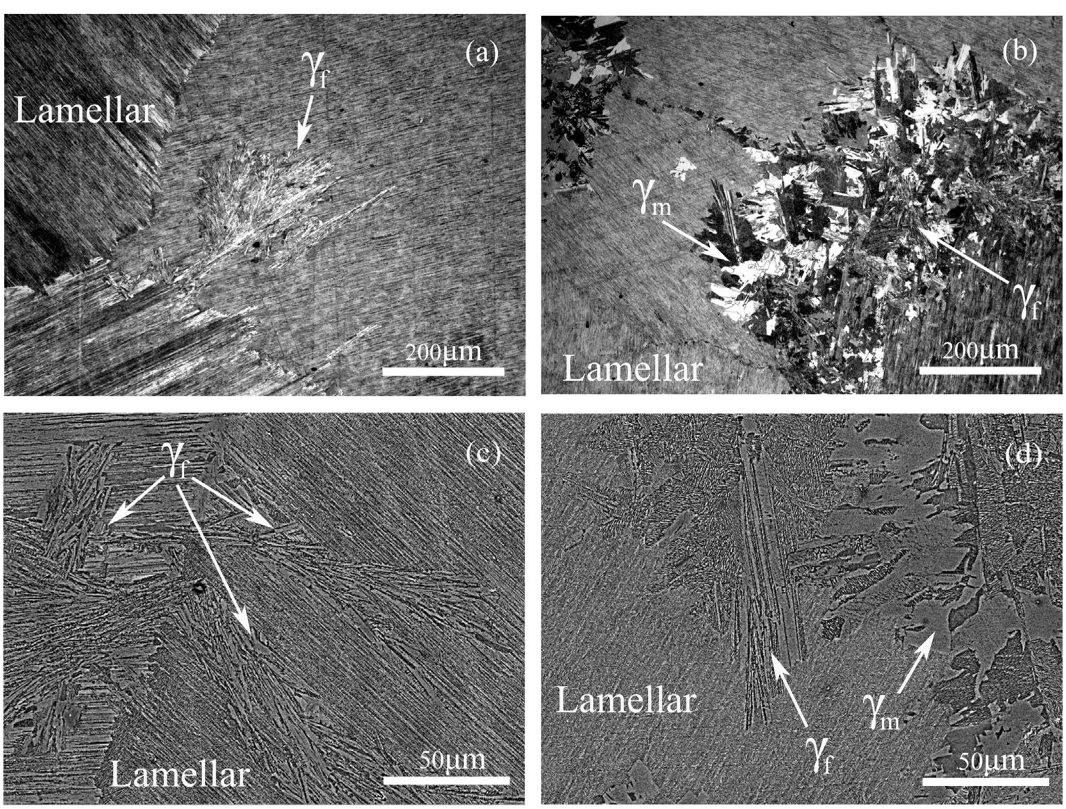

Fig. 2. (a) and (b) Optical micrographs of showing a mixture of γ_m, γ_f and lamellar microstructures in Ti48Al2Cr2Nb alloy at $\Delta T = 350$ K and $\Delta T = 370$ K, respectively and (c) and (d) back-scattering electron (BSE) images in the high magnification.